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Preliminary Evaluation of Adhesion Strength Measurement Devices for Ceramic/Titanium Matrix Composite Bonds

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Preliminary Evaluation of Adhesion Strength Measurement Devices for Ceramic / Titanium Matrix Composite Bonds

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Abstract:

The adhesive bond between a ceramic cement and a Titanium Matrix Composite substrate to be used in the National Aero-Space Plane program is evaluated in this study. Two commercially available adhesion testers, the Sebastian Adherence Tester and the CSEM REVETEST Scratch Tester, are evaluated to determine their suitability for quantitatively measuring adhesion strength. Various thicknesses of cements are applied to several substrates and the bond strengths are determined with both testers. The Sebastian Adherence Tester has provided limited data due to an interference from the sample mounting procedure, and has been shown to be incapable of distinguishing adhesion strength from tensile and shear properties of the cement itself. The data from the scratch tester have been found to be difficult to interpret due to the porosity and hardness of the cement. Recommendations are proposed for a more reliable adhesion test method.

Introduction:

The National Aero-Space Plane (NASP) will operate under high aerothermal loads. Sensors, such as temperature and strain, to be used on the NASP will require attachment techniques which are robust. In order to optimize the attachment procedures for these sensors, a method to measure the adhesion strength of the attachment material to the substrate must be developed. One of the substrate materials selected under the NASP program is

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a Titanium Matrix Composite (TMC). A flame sprayed 250 μm thick alumina coating is the candidate method and material used to attach a research strain gage to a TMC structure. The flame sprayed alumina will act as the electrical insulator and as the attachment material. Since this attachment procedure is new for TMC's, the purpose of this effort is to measure the adhesion strength of the alumina/TMC bond.

For a quantitative analysis of this adhesion strength, two commercial instruments are evaluated in this report. The first is the Sebastian Adherence Tester (SAT). The other is the CSEM REVETEST Automatic Scratch Tester. This report will document the performance of these two commercial units for measuring the bond strength between a porous ceramic cement and TMC.

In this report a manually applied ceramic adhesive is referred to as a cement. A ceramic applied by either the flame spray or plasma spray technique will be referred to as a coating. A ceramic cement is substituted in this study for the flame sprayed alumina coating. The adhesive or cement provides an insulating material which is inexpensive and easy to apply. The adhesive studies will optimize the substrate preparation method, ceramic cement thickness, and cement composition before submitting samples for flame spraying. Flame and plasma sprayed coatings will be investigated at length later in the program.

In order to evaluate the ability of these instruments to measure bond strengths of porous ceramic attachment materials, two studies have been designed. These studies involve two different sample preparation methods which have been selected in order to isolate instrumental variables from attachment material variables. The titles of these studies are listed below. The second study consists of two parts. The studies are

-Study I: Variable Cement Thickness on TMC

-Study II: Cement on Aluminum Foil Substrate

Part 1: Variable Cement Thickness on Aluminum Foil Substrate

Part 2: Determination of Depth of Fracture after Adhesion Test.

The variable cement thickness study involves using the SAT to measure the adhesion between TMC and cements of different thicknesses. These tests will assist in determining the optimal thickness for the tester and in determining if the properties of the cement itself influence the data.

The second study in which a smooth aluminum foil substrate is substituted for the TMC is designed to answer two questions that have arisen while using the SAT. First, as a result of a better understanding of the SAT's operation, it is questioned whether the instrument is really testing adhesion between the cement and the substrate or if it is tensile and shear testing the cement only. The second question requires a brief explanation (see Figure 1). The SAT is a tensile pull tester that uses a stud which is bonded to the cement with an epoxy. Does the epoxy diffuse through the porous cement during the curing process and bond itself to the substrate? If the answer is yes, results from the test would be invalid since the tester would be measuring the adhesion between the epoxy and substrate rather than the adhesion between the cement and substrate (TMC).

In an effort to answer the above questions, part one of the aluminum foil substrate study involves preparing variable thickness ceramic cement samples on a substrate that will give little to no backing strength. The aluminum foil substrate is expected to tear during the test and contribute little to the load to failure value. This set of experiments is an attempt to remove the adhesion factor from the test result and test only the tensile and shear factors. If adhesion, rather than tensile strength, is a major factor in the test, a significantly lower load to failure value is expected for the aluminum foil substrate samples as opposed to that of the TMC substrate samples due to the lower backing strength of the aluminum foil.

In part two of the aluminum foil study, the fractured surfaces of the samples from part one will be further analyzed to determine the depth of fracture or depth of epoxy penetration into the ceramic cement. All samples will be tested using the same procedure as for the variable thickness studies on TMC's. Results from the aluminum foil substrate test will be compared to data from TMC substrate studies.

For a preliminary investigation of the scratch tester TMC substrates are coated with the ceramic cement. After analyzing the initial results, recommendations are proposed for more reliable scratch test measurements.

Experimental Procedure:

A.) General Sample Background:

The substrate materials included TMC and aluminum foil. The TMC was a SiC/Ti - 24Al - 11Nb(at. %) which was obtained from the Materials Division at NASA Lewis Research Center. Two different surface finishes were applied to the TMC's by polishing some with 150 grit SiC paper and others with 600 grit SIC paper to aid in determining if mechanical bonding is a factor in adhesion testing. The aluminum foil was 50μm thick and oil free from All Foils, Brooklyn Hts., Ohio. The aluminum foil was used as received with no further preparation.

For all studies Cotronics No. 903HP cement was used. This cement was a 3000°F water based alumina adhesive that was mixed thoroughly and manually applied with a steel spatula. The edge of a glass slide was then used to smooth the cement to the proper thickness. The resulting thickness was fairly uniform ($\pm$ 50μm) from sample to sample if the slide position and tilt were maintained as constant as possible by the laboratory person.

The adhesive curing process for 903HP began with a 24 hour exposure in air at room temperature. This was followed by placing the samples in a Fisher Isotemp Programmable Ashing Furnace (model 495) which was programmed to increase oven temperature at a rate of 5°C per minute to a holding temperature of 121°C for 2 hours. The oven temperature was then increased at a rate of 10°C per minute to a holding temperature of 371°C for 4 hours. Finally, the samples were allowed to cool gradually back to room temperature in the oven with the door closed.

All thicknesses were measured using a Heidenhain VRZ 401 Bidirectional Counter which is a thickness tester with a resolution of 0.5 μm. Since the powder metallurgy manufacturing process used to produce TMC's resulted in a non-uniform surface, an average value was taken from several positions on the TMC surface area surrounding the cement. The sample was moved to measure the cemented area, where another set of measurements was averaged. The thickness of the substrate and cement (T_{S+C}) minus the thickness of the substrate (T_S) results in the approximate thickness of the cement (T_C).

$$T_{S+C} - T_S = T_C \qquad \text{Eq. 1}$$

Study I: Variable Cement Thickness Sample Preparation:

In the variable thickness study, the alumina cement was manually applied to (12.5mm x 12.5mm x 0.75 mm) TMC substrates, polished to a 600 grit finish with SiC paper. The initial cement thickness was 250 μm. One half of the cemented area was then thinned to 12 μm with 320 grit SiC paper. The surface of the 250 μm thick section was smoothed slightly to obtain a similar surface roughness on both the thick and thin cements. Only five TMC samples were prepared and tested, due to the limited availability of TMCs.

Study II: Cement on Aluminum Foil Substrate Sample Preparation:

Part 1:

For the foil substrate study, thirty 903HP cement samples of thicknesses varying from 250 μm to 1250 μm were manually prepared using the shiny side of 50 μm thick aluminum foil as a substrate. Prior to the application of the cement, the aluminum foil was wrapped around a glass slide (5cm x 15cm) to add stability to the foil and prevent it from curling during the heat treatment required to cure the adhesive. After curing the cement applied to the aluminum foil samples, the glass slide backings were removed, and the excess foil was cut away from around the cemented area. The aluminum foil was removed altogether before testing two of the samples in order to verify the minimal backing support of the foil. This verification would be achieved by comparing the results of the samples without a substrate to the samples with a substrate. Following the sample preparations, the bond strength or adhesion strength was measured using the SAT.

Part 2:

In the second part of the foil substrate study, samples from part 1 above which had the highest load to failure values were further analyzed to determine the depth of fracture. The depth was measured using the Heidenhain VRZ 401 Bidirectional Counter. In this measurement the average cement thickness surrounding the fracture (T_{co}) minus the average cement thickness at the point of fracture (T_{cf}) equals the depth of fracture (T_f).

$$T_{co} - T_{cf} = T_f \qquad \text{Eq. 2}$$

B.) Adhesion Test Methods:

SAT-Sebastian Adherence Tester

This adhesion tester was a tensile pull tester that used a 2.50mm diameter pull stud that was bonded to the adhesive cement with an epoxy. During the 150°C, 1 hour epoxy cure, the pull stud was physically clamped to the sample with a spring clip. The spring clip maintained pressure between the pull stud and the cement surface and also kept the stud perpendicular with respect to the sample surface. It was observed that the spring clip pressure varied from clip to clip. After curing, the spring clip was removed.

The pull stud with the sample attached was placed in the SAT, where a tensile load was applied by the instrument to the pull stud resulting in its removal from the test sample. A cross sectional view of the SAT is shown in Figure 1. The load at which the bond fails was recorded as load to failure in PSI which should be a measure of bonding strength.

CSEM REVETEST Automatic Scratch Tester

For scratch tester experiments a CSEM REVETEST Automatic Scratch Tester (Neuchatel, Switzerland) was used. In this system a 12.5mm x 12.5mm TMC coated with 250 µm of cement was clamped to the testers table. The table moved 12mm in one direction during the test. During the test the sample surface was scratched with a diamond tip stylus subjected to a progressively increasing load ranging from 0 to 100 N (Newtons). Figure 2 depicts a simplified schematic diagram of the operation. With the stylus coupled to a transducer, an acoustic emission (AE) resulted from the scratch, similar in concept to that of a phonograph needle. The instrument recorded the AE vs load. A change in the AE would be generated as the stylus scratched through the cement/substrate interface. The stylus load at this interface was usually an AE transition point, and a measure of the critical load (in N) for the adhesive. With the aid of the microscope attached to the instrument, the critical load of the adhesive could also be determined visually by measuring the distance the stylus traveled until the substrate was initially contacted (exposed). The attached counter produced a readout of distance in mm. Since the load was progressive, the critical load (C_L) could be calculated by dividing the distance traveled by the stylus to substrate contact (d_S) by the maximum distance traveled by the stylus during the test (d_T).

$$(d_S / d_T) \cdot 100N = C_L \qquad \text{Eq. 3}$$

<u>Results:</u>

After limited tests, the results from the variable thickness ceramic cement study on TMC using the SAT are recorded in Table 1 which lists the surface finish, cement thickness, and load to failure values of the samples tested. In Figure 3 the load to failure (in PSI) values recorded in Table 1 are plotted as a function of cement thickness. The two 12 µm thick cements result in high (>100 PSI) load to failure values. The three 250 µm thick cements yield low (<100 PSI) values. From these results, the thick cements tend to break within the cement, with little or no load. The thinner cements are more difficult to delaminate, with breakage occurring at the substrate/adhesive interface.

For the aluminum foil substrate testing, a much larger number of samples are tested using the SAT. Table 2 lists the cement thickness, load to failure, and depth of fracture values. In Figure 4, the thickness is plotted as a function of load to failure (in PSI). Note the weak linear relationship exhibited by the strongest bonds. These results show that the thicker cements are more difficult to delaminate. This relationship is opposite of that observed in the previous experiment (Figure 3). This particular study uses an aluminum foil substrate to provide little backing strength at the adhesive/substrate interface and is designed to remove the adhesion factor altogether. Since adhesion between the cement and the substrate should not be influencing the data in Figure 4, these results suggest that factors other than interface bond strength are involved in this test method.

In an attempt to explain the increasing load to failure values with increasing thickness, the samples with the highest values in Figure 4 are further analyzed to determine if different amounts of cement are removed at failure. If more material is removed or the depth of fracture is greater, the result may suggest that epoxy diffusion into the cement increases. The effective area to which the load is applied increases as epoxy penetration increases. Thus a larger area (increased depth of fracture) can support a larger load, as shown in Figure 4.

Figure 5 highlights the samples which exhibit the highest load to failure values. The results show greater fracture depth with increasing cement thickness. A plateau occurs in the graph at a thickness of 500 μm which may be the maximum depth to which the epoxy may penetrate due to the uniform amount of epoxy used on each pull stud.

Discussion:

A.) SAT-Sebastian Adherence Tester:

From the results shown in Figures 3 - 5, the aluminum foil substrate experiments provided substantial information to aid in understanding the operation of the SAT and its use with these materials and attachment methods. Data from the aluminum foil substrate experiments provided information useful in the interpretation of the variable thickness ceramic cement data. Further discussion of the variable thickness ceramic cement studies will follow the aluminum foil substrate discussion below.

In the aluminum foil substrate study, part one, load to failure values of less than 100 PSI (Figure 3) are attained. All testing done to date with ≈250 μm thick 903HP cement has resulted in load to failure values of less than 100 PSI, regardless of the substrate used, as summarized in Tables 1 and 2. Because of the fact that different substrates have little effect on the adhesion test results, it is concluded that adhesion is not a factor in these tests. Further evidence of this conclusion is obtained from cements thicker than 500 µm in which the failure took place within the cement itself, never reaching the foil.

When a bond failure occurs within the cement, adhesion to the substrate cannot possibly be a factor. The failure is a result of the tensile and shear loading of the cement only. Since the results of the TMC substrate, the aluminum foil substrate, which has little backing strength, and the two cement samples with no substrate and no backing strength are comparable (<100 PSI) for all samples tested on the SAT, they support the idea that the instrument is only testing tensile and shear strength, not adhesion.

Additional support for this idea comes from visual observations made during the foil substrate testing. Both the actual sample and the numerical value of the load can be monitored simultaneously by the operator. The instrument increases the load until the shear strength of the cement is exceeded. At this point the foil can be seen to dimple inward as the cement is further pulled down. No additional load is recorded by the instrument even though the cement continues to peel away from the foil. This suggests that the adhesion between the foil and the cement is minimal and not within the resolution of this equipment. Since the adhesion factor has been shown to be removed from the test it is concluded that the instrument and is only measuring the shear and tensile strength of the ≈250 µm thick cement used in this project.

In part two of the aluminum foil substrate study, the results show that the samples with the greatest depth of fracture resulted in the highest load to failure values. These data suggest the idea that epoxy penetration may influence the test. Since the epoxy, which is inherently less brittle than the cement, tends to flow into the porous cement, the epoxy will strengthen the cement. As the epoxy penetration increases, the area at which the load is applied also increases, thus the load is spread over a larger area and more load can be withstood by the cement. By measuring the depth of fracture of the samples, one verifies that epoxy diffusion can take place to a depth of 500 µm.

Spring clip pressure may account for a part of the increased epoxy diffusion. A greater pressure between the pull stud and the cement during the epoxy curing process may cause the epoxy to flow more readily and deeper into the porous cement. As cement thickness is increased, spring clip pressure is also increased, due to increased spreading of the clip, thus increasing the epoxy penetration potential. The effects of this statement can be seen in Figure 5 where load to failure values are the highest with the thickest cement.

It is now believed that the results from the variable cement thickness study are due to epoxy penetration into and in some cases through the cement, and not from the reduced cement thickness. It has been seen in preliminary testing that epoxy bonded directly to TMC can exceed a 10,000 PSI load to failure value, thus the epoxy can add considerable strength if it contacts the TMC. Thinning the cement enhances epoxy diffusion to the substrate and is responsible for the increased values of Figure 3 The difference in these two values (≈500 & 2000 PSI) is believed to be due to the variable density of the cement.

The porosity of the cement allows the epoxy to reach the substrate in our cement studies. The question arises whether the porosity of the flame spray and plasma spray coatings would result in a similar problem for the SAT test. Scanning Electron Microscope (SEM) micrograph studies of the cement, flame spray, and plasma attachments, (not shown in this report) indicate that all are porous. The approximate size of the pores (at the surface) of the flame spray and plasma spray coatings is comparable to those pores in the cement's surface. On this basis, it is believed that the same difficulty (epoxy diffusion) will occur with the SAT testing of the flame spray and plasma spray coatings.

The results discussed above indicate that the adherence tester (SAT), when used with porous ceramic cements, primarily tests the tensile and shear strength of the cement and not the ceramic adhesive/substrate bond strength. To further expand on this point, the adherence tester's operation is discussed as follows. During the test, the material above the platten is subjected to a compressive force, the material above the stud is placed in tension and the material between the stud and the platten is placed in shear. The shear force of the adhesive material itself must be exceeded before adhesion between the adhesive ceramic and the substrate actually becomes a factor. With this instrument, one cannot differentiate among the three forces (shear, tensile, and adhesion). Only the maximum force applied at the time of failure is recorded. Therefore, the results of the test are a combination of the three forces and not a true indication of adhesion only. Due to the inherently low tensile and shear strength of a ceramic, an accurate measurement of adhesion cannot be made in this application with the SAT. The instrument is therefore not suitable for use in this project.

B.) CSEM REVETEST Automatic Scratch Tester:

Preliminary work with the scratch tester has resulted in the following. The acoustic emission data have been found to be very difficult to interpret. A hard cement being placed atop a hard substrate produces indistinguishable changes in the AE curve. The emission curve does not appear to change at the transition point as expected, to indicate bond failure. The porosity and roughness of the ceramic cements may be a factor.

Figure 6 is a typical AE curve that has been obtained for these adhesive cements on TMC. The extremely high noise level which is most likely attributed to the roughness of the cement obscures any transition point.

The scratch tester may be useful for qualitative data used to compare one cement to another by visually measuring the distance traversed by the stylus until the substrate is contacted. This instrument may also be useful with thin film gage installations which are in the range of 2 to 8 μm thick, and also in which the cement porosity and roughness are not large factors.

Conclusions:

It is concluded that the Sebastian adherence tester (SAT) is not suitable for use with the porous ceramic cements used in this project. The two major problems with this tester are the following:

1.) Epoxy penetration of the pull stud attachment during curing.

2.) Inability to distinguish adhesive strength from tensile and shear forces .

The instrument has been found to measure tensile and shear stress of the cement which unfortunately is not of interest in this application. The epoxy diffusion factor will continue to be a problem with the flame spray coatings to be used in the future, because these coatings are also porous.

The scratch test AE data are found to be hard to interpret due to the physical structure of the samples used. The test may be of value in ranking one adhesive against another by using the optical examination technique. Its most valuable use may be for smooth, dense, thin film coatings prepared by sputter deposition. Further investigation of this instrument will be initiated with the thin film sensors.

The literature search conducted failed to produce a quantitative test method suitable for this application. Most tests reviewed were primarily qualitative, or not feasible by design for flame spray application techniques. The methods consisted of tensile pull, lap shear, scratch, or tape tests. Therefore, a more quantitative test apparatus is being designed and built according to the plans described below.

Proposed Further Studies:

Because of the problems with the instruments currently available, it is proposed that a Horizontal Pull Adhesion Tester be built in house. (see Fig. 7) This system will be similar to the prototype Cement Pull Test Apparatus developed at HiTech Products, Inc. Ayer, MA 01432. The HiTech device is used to test lead wire attachments applied with ceramic cements. The operating principle is as follows. The sample with ceramically bonded lead wires is clamped to a slide capable of moving in the X direction. The lead wire is attached to the free end of a cantilever beam which has two commercially available low temperature strain gages attached to its sides. The slide is moved to apply a tensile load to the lead wire, the actual load applied is calculated from resistance measurements of the strain gages attached to the bar. The wire is pulled to failure and the load at which this occurs is recorded.

In the proposed plan, a modification of the experiment will consist of embedding a 75µm diameter Nicrosil wire into the cement, then applying a horizontal tensile load (parallel to the sample surface) that hopefully removes the cement from the substrate in one complete piece. The load at which this occurs will be calculated from resistance measurements. The instrument will be calibrated using dead weights. Nicrosil wire was selected on the basis of its high tensile strength at temperatures up to 1000°C. This wire is currently used in high temperature strain gage development.

Some parameters are still variable. One is the geometry of the wire. The shape into which the wire is pre-formed should be designed to keep the stress imposed on the cement due to the presence of the wire to a minimum. At this time a flattened wire formed into a loop with a spot welded end seems to be the best candidate. The loop should uniformly spread the load over a large area and place some of the ceramic in the center of the loop under a compressive load. This position is advantageous because ceramics typically have high strength in compression. The appropriate depth of which the loop is placed in the cement must also be determined.

Once a wire geometry is optimized, wires will be formed using a jig to assure uniformity. The rate of load application will be identical for every test.

The horizontal pull adhesion tester will more accurately simulate conditions to which the gage will be subjected under actual operating conditions. It is suspected that cement porosity will not be a problem as with the epoxy pull stud test (SAT). In this application, the wire will be anchored by the ceramic cement itself. A limitation with this instrument will be that it requires cement applications of 250 μm thick or greater, but these thicknesses are comparable to flame sprayed ceramic attachments.

In future work the plasma spray and flame spray application method will be utilized for applying ceramic coatings for test purposes. These two methods are preferred by industry and are currently used in the fabrication of strain gages.

Because of low TMC availability and limited knowledge of its properties, a super alloy, Inconel, will be used as a baseline for comparison to TMC. Inconel is readily available, costs less and is widely used in the aeronautics industry.

Table 1: Sebastian Adherence Test Results of
903HP Alumina on TMC

SiC / Ti -24Al -11Nb (at%)

TMC Finish	Cement Thickness in µm	Load to Failure PSI
600 SiC	250	90
600 SiC	12	470
600 SiC	12 R	1920
600 SiC	225	80
150 SiC	250	90

R = repeat study

The number of test results shown here were limited by TMC availability.

Table 2: Sebastian Adherence Test Results of
Cotronics 903HP Alumina bonded to Aluminum Foil

Sample	Cement Thickness (μm)	Load to Failure in PSI	Depth of Fracture (μm)
None 1	800	22	
None 2	675	40	400
Foil 1	325	Invalid	
Foil 2	575	0	
Foil 3	400	17	
Foil 4	675	1	
Foil 5	400	23	340
Foil 6	325	13	245
Foil 7	675	18	
Foil 8	600	38	358
Foil 10	1175	81	530
Foil 11	700	12	
Foil 12	850	0	
Foil 13	500	31	403
Foil 14	450	5	
Foil 15	550	7	
Foil 16	875	63	465
Foil 17	650	19	
Foil 18	475	18	
Foil 19	350	12	
Foil 21	250	40	E
Foil 22	250	20	E
Foil 23	250	60	E
Foil 24	250	20	E
Foil 25	250	30	E
Foil 26	250	20	E
Foil 27	250	10	E

E = Foil exposed after test

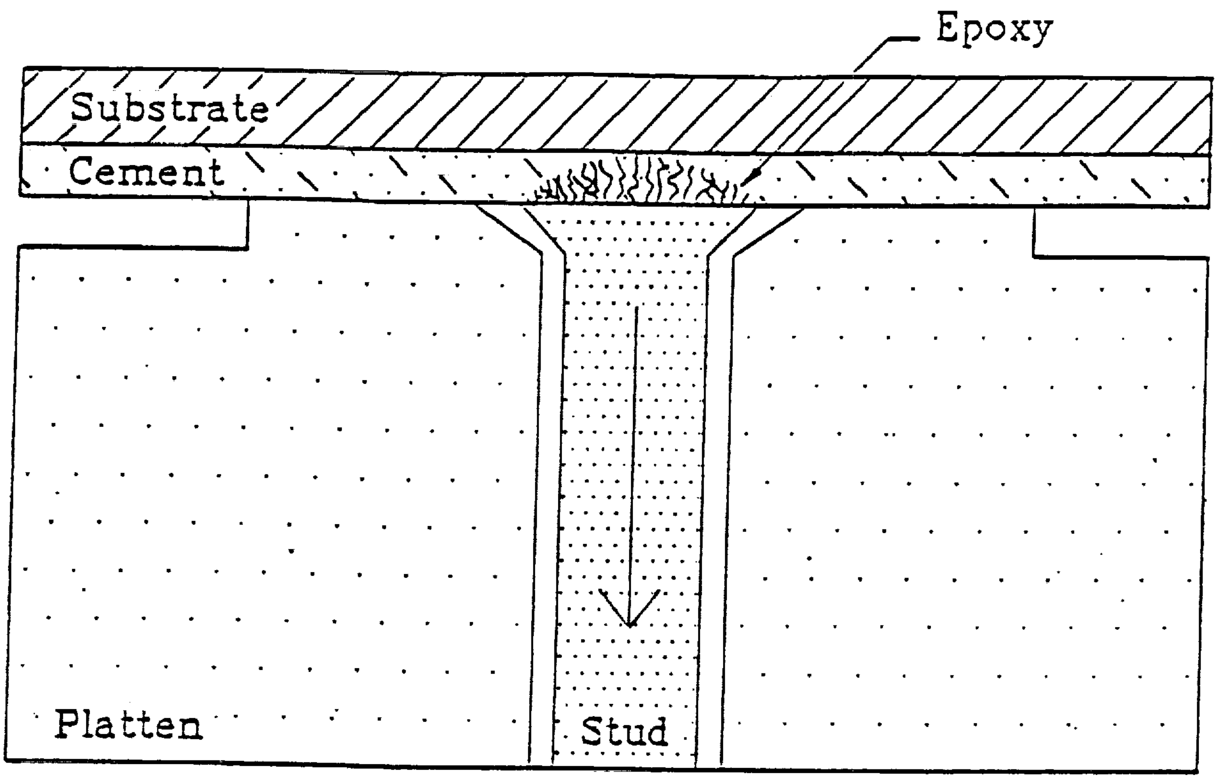


Figure 1: A cross sectional view of the Sebastian Adherence Tester. The 2.50mm diameter pull stud is bonded to the cement surface with an epoxy that cures at 150C for 1 hour. The pull stud is then clamped into the instrument which applies a tensile load to the stud, resulting in its removal from the sample. The load at which this occurs is recorded as Load to Failure in pounds per square inch (PSI).

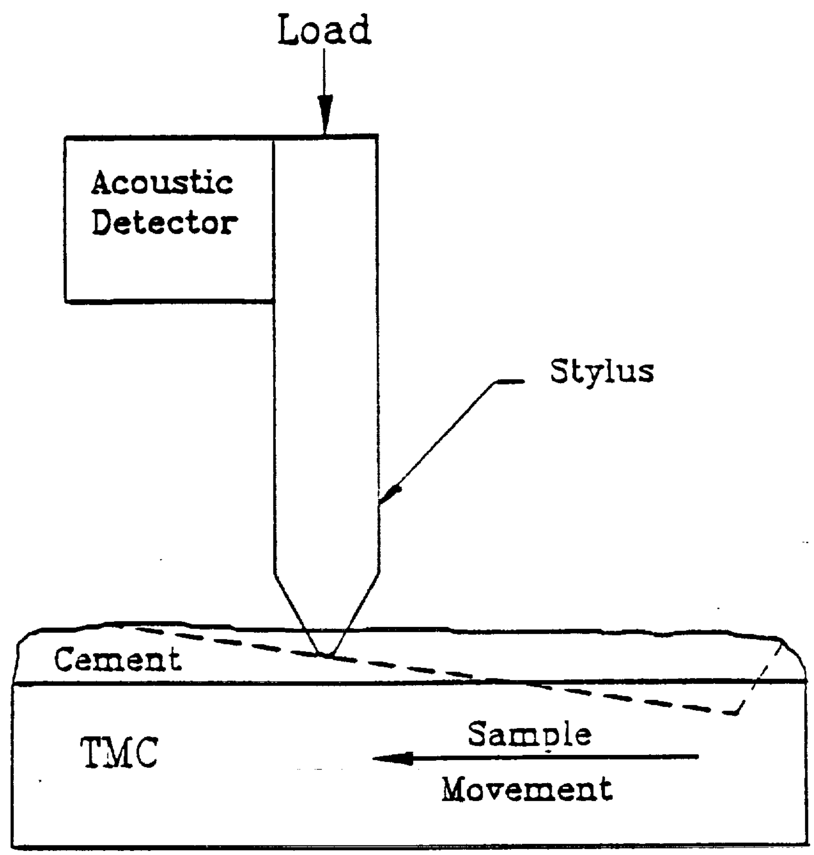


Figure 2: A simplified schematic view of the scratch tester. The stylus is subjected to a progressively increasing vertical load, while the sample moves horizontally beneath it. An Acoustic Emission is generated by the action of the stylus being pushed into the cement, thus setting up a stylus vibration that is recorded by the detector.

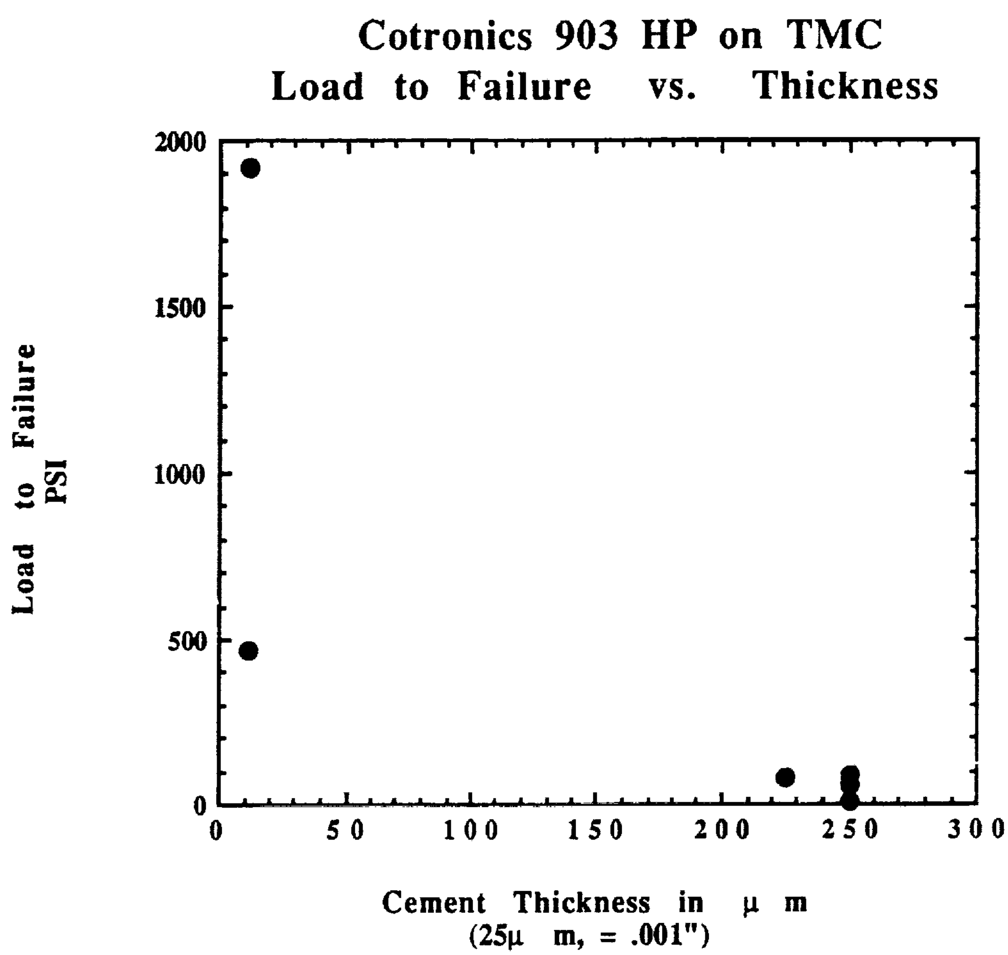


Figure 3: Load to Failure values obtained with the Sebastian Adherence Tester and summarized in Table 1 are plotted as a function of Cotronics 903HP Alumina cement thickness. The substrate is TMC.

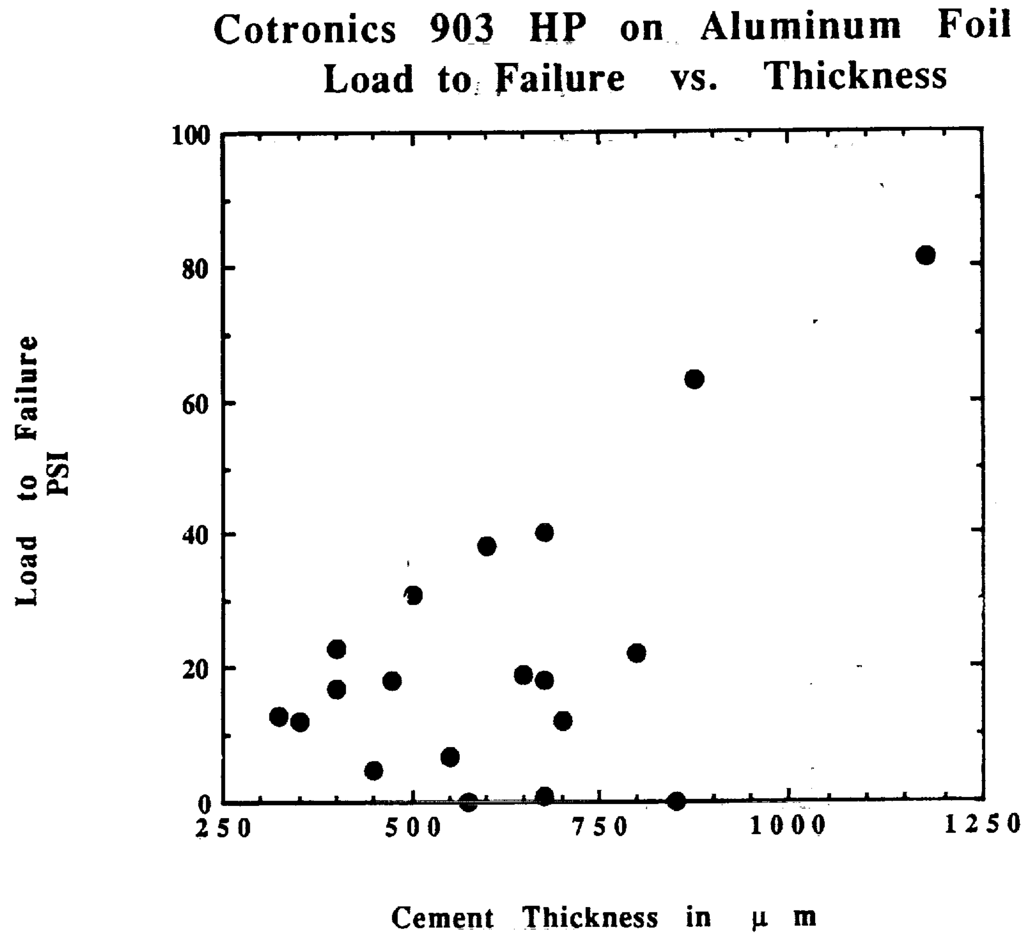


Figure 4: Load to Failure values obtained with the Sebastian Adherence Tester of the samples in Table 2 which did not expose the aluminum foil substrate are plotted as a function of cement thickness.

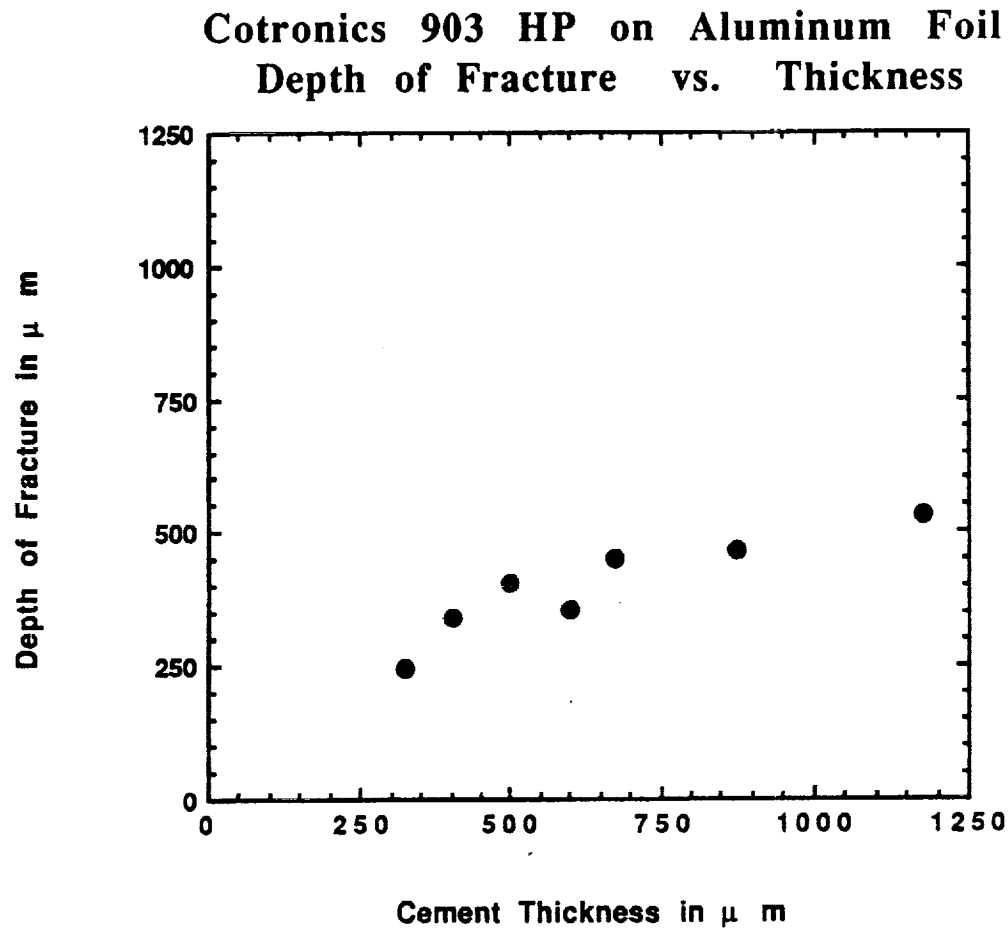


Figure 5: Depth of fracture values obtained as a result of the Sebastian Adherence Test are plotted against cement thickness. The samples with the highest load to failure values from Figure 4 are shown here.

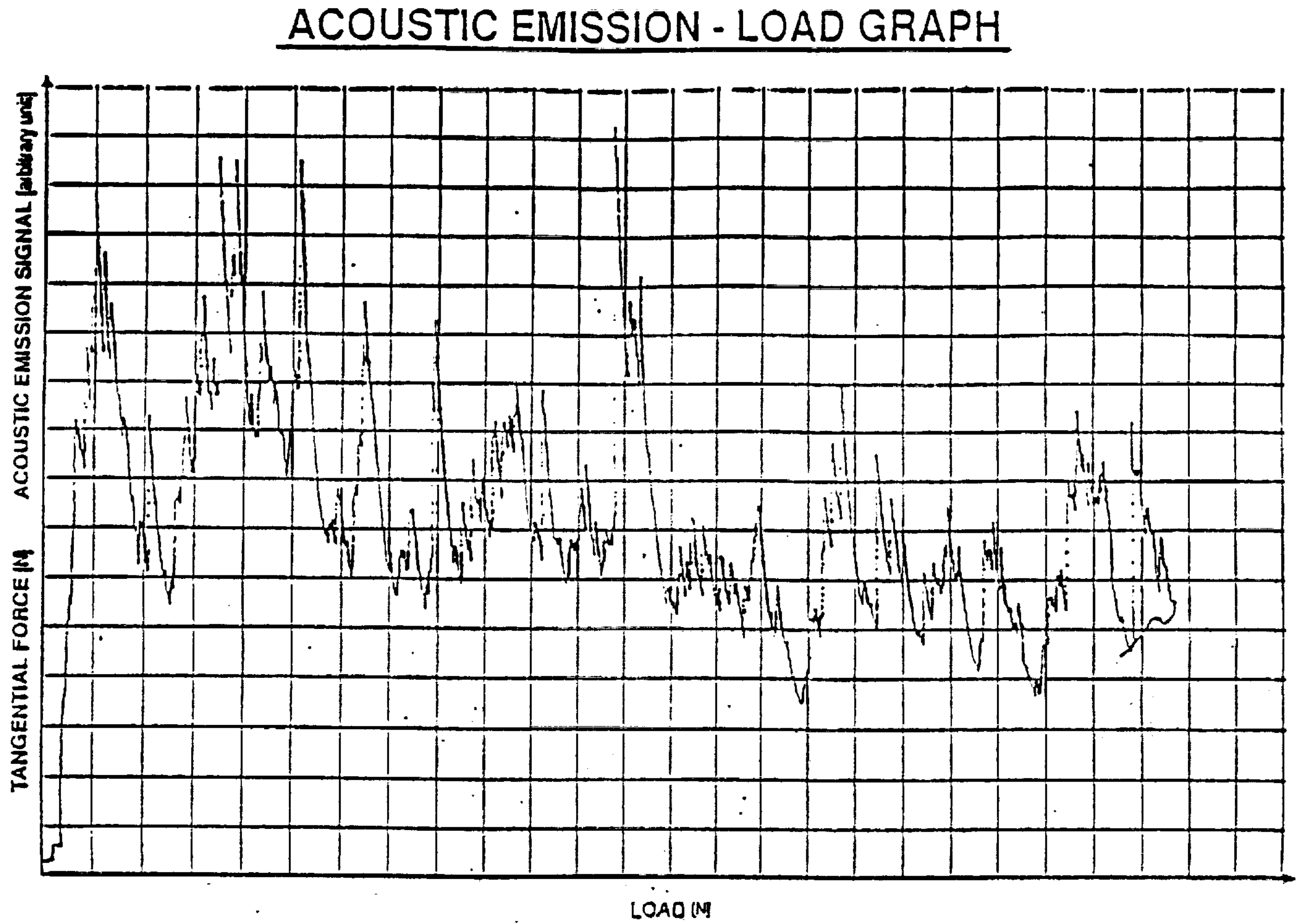
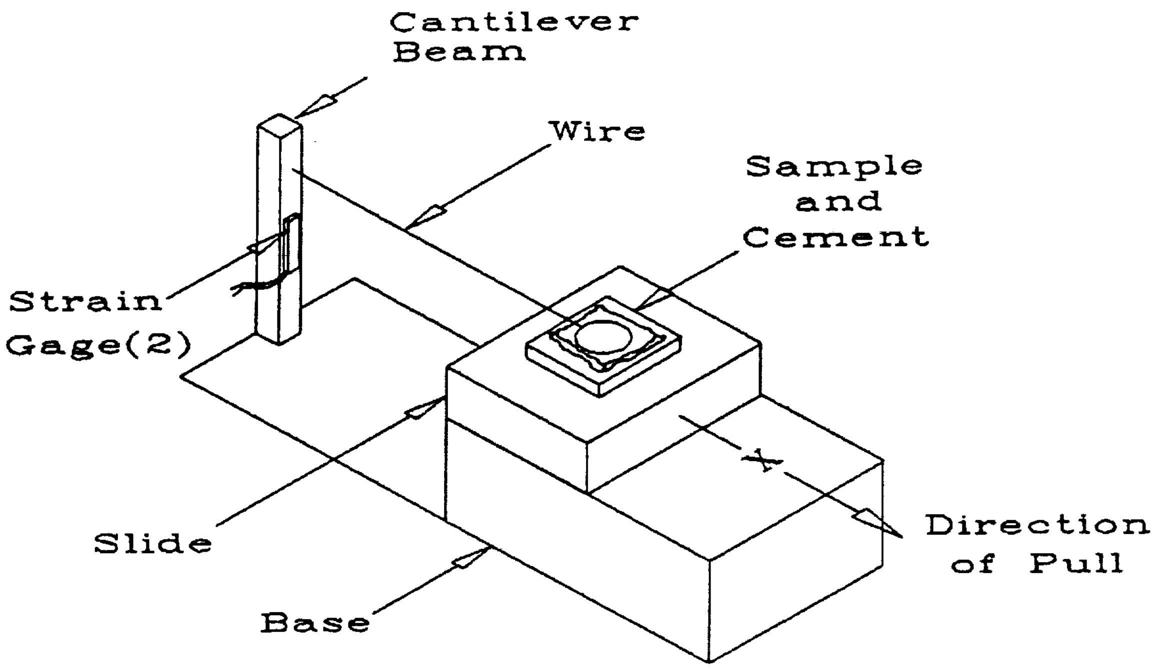


Figure 6: An example of Acoustic Emission vs. Load data resulting from a scratch test of a ceramic cement bonded to TMC.



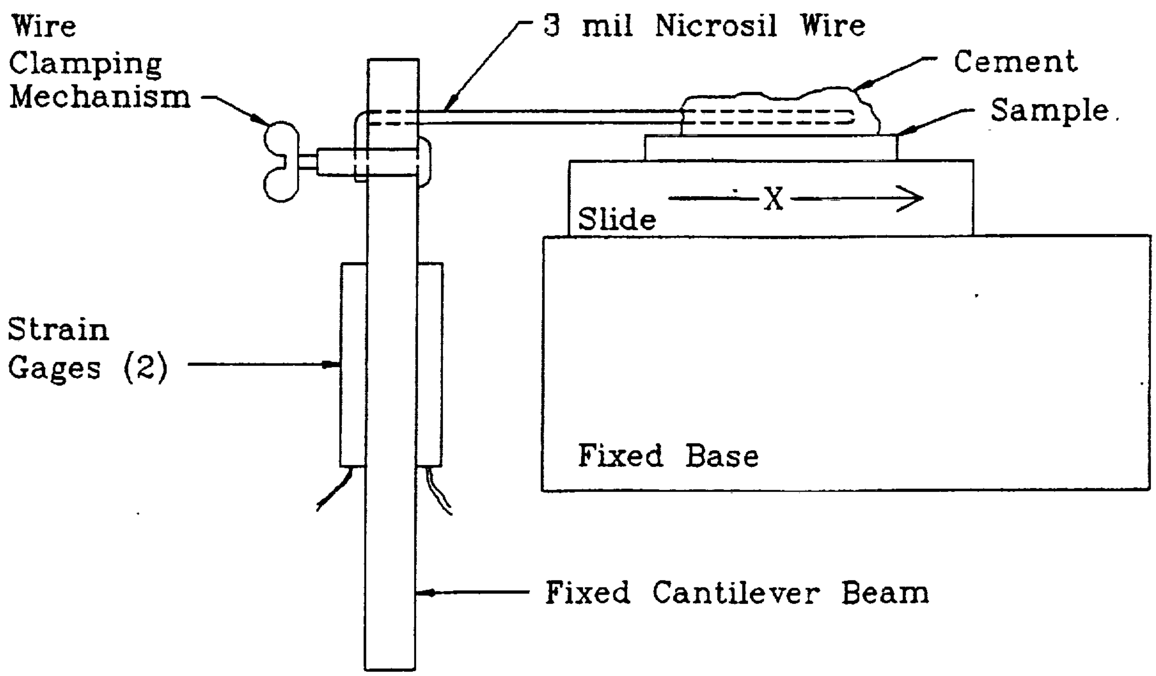


Figure 7: An simplified isometric and detailed side view of the proposed horizontal pull adhesion tester that is to be built in house.

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13. ABSTRACT ***(Maximum 200 words)***

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